

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

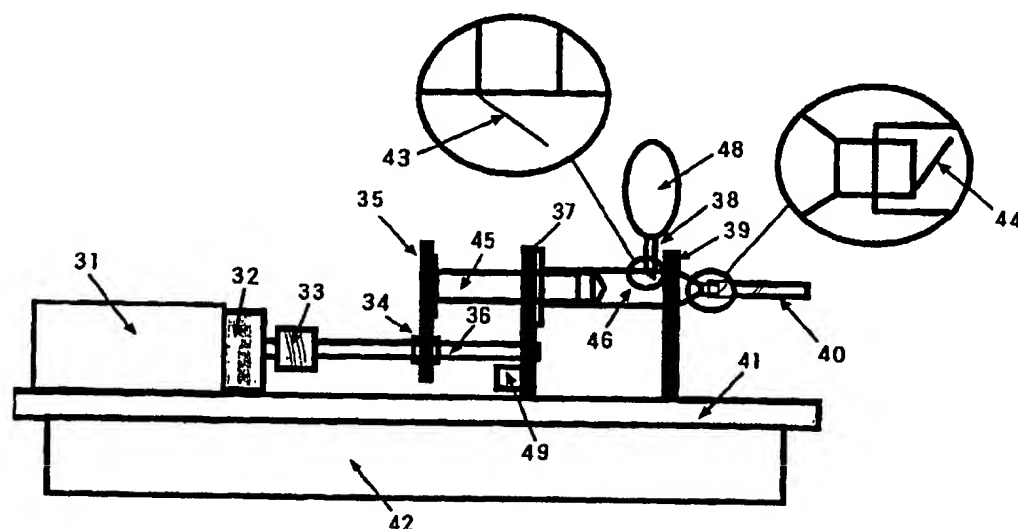


(43) International Publication Date
27 September 2001 (27.09.2001)

PCT

(10) International Publication Number
WO 01/70281 A1

- (51) International Patent Classification⁷: A61L 2/14, 2/18, 2/24
- (21) International Application Number: PCT/KR00/00539
- (22) International Filing Date: 26 May 2000 (26.05.2000)
- (25) Filing Language: Korean
- (26) Publication Language: English
- (30) Priority Data:
2000/14750 23 March 2000 (23.03.2000) KR
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- (54) Title: PLASMA DISINFECTION SYSTEM
- (51) International Patent Classification⁷: A61L 2/14, 2/18, 2/24
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- (81) Designated States (national): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:
— with international search report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



(57) Abstract: There is provided a low temperature plasma sterilization system comprising a reaction chamber (1) for receiving an item (9) to be sterilized, said item being wrapped in a packaging material (10); an anode (2) and a cathode (3) disposed above and below the item (9) to be sterilized in the reaction chamber (1) respectively; an injection heater (5) connected to said anode (2) via a mass flow controller (4); a plasma power source (8) connected to said cathode (3) via an impedance matching circuit (6) and an impedance matching controller (7) for generating high-frequency power; and a vacuum pump (11) disposed at the lower part of said reaction chamber (1), wherein a hydrogen peroxide solution (12) in the liquid phase is turned into gaseous hydrogen peroxide by means of said injection heater (5) prior to the sterilization with plasma, and then the gaseous hydrogen peroxide is adjusted and injected at desired pressure by means of said mass flow controller (4), to accomplish the pre-treatment.

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TITLE

PLASMA DISINFECTION SYSTEM

TECHNICAL FIELD

The present invention relates to a plasma disinfection system for sterilizing and
5 disinfecting microorganisms on surfaces of objects such as medical instruments with
gaseous plasma, and more particularly to a plasma disinfection system in which hydrogen
peroxide is supplied by extremely small fixed quantities as active species during
generation of the plasma and a pre-treatment is also carried out with gaseous plasma
prior to the generation of the plasma.

10

BACKGROUND ART

Various methods of sterilization and disinfection have been used for the
sterilization of different types of disposable and reusable medical equipments. Among
these methods, a method of sterilization and disinfection by steam or by dry heat
has been used extensively used. However, this method of sterilization and disinfection
15 is not useful to sterilize materials that are adversely effected by such heat or steam.

Ethylene oxide (EtO) gas has also been used but suffered from the drawback
that it may leave toxic residues on the articles to be sterilized, which may have adverse
effects on patients who come into contact with such articles. Consequently, with this
method, the extended aeration cycles required to remove residual ethylene oxide from
20 some sterilized items also makes ethylene oxide sterilization excessively long.

In order to overcome the aforesaid drawback, hydrogen peroxide, which has
been used widely as a disinfectant, has been used. Hydrogen peroxide has been known
to have bactericidal properties and has been used in solutions to kill bacteria on surfaces
on various articles to be sterilized.

25

U.S. Patent No. 4,437,567 discloses the use of aqueous hydrogen peroxide

solutions at low concentrations, i.e., 0.01 % to 0.1 % by weight, to sterilize packaged products for medical or surgical use. However, with the method of this patent, sterilization requires at least fifteen days at room temperature, and sterilization can be accomplished in approximately one day at higher temperature. In other words, it is required to heat
5 the aqueous hydrogen peroxide solutions at higher temperature in order to accomplish more rapid sterilization.

U.S. Patent No. 4,169,123, No. 4,169,124, and No. 4,230,663 disclose the use of hydrogen peroxide in the gas phase at temperature below 80°C and concentrations of 0.1 to 75 mg H₂O₂ vapor/liter to volume of a reaction chamber for sterilization
10 and disinfection.

The use of ultraviolet (UV) radiation with hydrogen peroxide for improved antimicrobial activity has been disclosed in U.S. Patent No. 4,366,125 and No. 4,289,728. With the methods of these patent, surface of the object to be sterilized can not be directly exposed to the UV radiation because of the package in which the object to
15 be sterilized is placed, resulting in that the object in the package can not be sterilized.

An apparatus for injecting hydrogen peroxide in the plasma sterilizer has been used. A capsule type cassette system containing a prescribed quantity of hydrogen peroxide (for example, 10 pieces) in it has been employed in the ASP Model of Johnson and Johnson which manufactures and sells the low temperature plasma sterilizer.
20 In the capsule type cassette system, the solution contained in the capsule is supplied into the solution feeding pipe by means of the injection pump, and the supplied hydrogen in the liquid phase is vaporized in the vaporizer, which is then fed into the sterilization reactor.

With the aforesaid capsule type cassette system, however, the used cassette
25 must be replaced by a new one with 10 pieces in it after the sterilization process is carried out ten times since one capsule is used in one sterilization process.

In addition, a vaporizer with a liquid mass flow controller and an injection

pump is used broadly to supply liquid for generation of vapor of the metal into the chemical reaction chamber where the plasma chemical reaction occurs in the metal organic chemical vapor deposition process in which a metal film used the most as a pattern circuit of semiconductor.

- 5 However, the aforesaid vaporizer has drawbacks that the apparatus for supplying an extremely small fixed quantity of the liquid is very complicated and the costs for manufacturing it is extremely high.

DISCLOSURE OF THE INVENTION

- 10 It is an object of the present invention to provide a plasma disinfection system which employs hydrogen peroxide as a precursor of the active species and provides an initial contact of surfaces of the material to be sterilized with the gaseous hydrogen peroxide injected into a reaction chamber as a pre-treatment step before the generation of plasma at a power level sufficient to achieve sterilization, resulting in that the total time and power required to accomplish sterilization with the low temperature
- 15 plasma, the use of the pre-treatment with hydrogen peroxide also allows sterilization to occur within many different types of packaging material, and no toxic residues remain on the sterilized items after plasma treatment since the decomposition products of hydrogen peroxide in plasma, which is obtained when final sterilization is carried out with the hydrogen peroxide plasma generated by the application of the power
- 20 after the pre-treatment, include water, oxygen and hydrogen.

 It is another object of the present invention to eliminate any inconvenience caused by frequent replacement of cassettes as in the capsule type cassette system and reduce the manufacturing costs by simplification of the structure.

- 25 It is still another object of the present invention to provide an apparatus for supplying an extremely small fixed quantity of liquid for injection, which may be used in hospitals and an apparatus for supplying an extremely small fixed quantity

of liquid with a vaporizer, which may be used to supply a fixed quantity of the liquid for generation of vapor of the metal used the most in the semiconductor process into the chemical reaction chamber, said apparatus being used to supply and vaporize an extremely small fixed quantity of hydrogen peroxide in the liquid phase used the
5 most for generating plasma for sterilization in the low temperature sterilization system or to supply and vaporize an extremely small fixed quantity of the metal organic compound in the liquid phase for generating the metal vapor plasma in the chemical reaction chamber under the stable condition in the semiconductor process for depositing the metal film using the metal organic chemical vapor deposition process.

10 To accomplish the aforesaid objects, one aspect of the present inventions provides a low temperature plasma sterilization system comprising: a reaction chamber for receiving an item to be sterilized, said item being wrapped in a packaging material; an anode disposed above the item to be sterilized in the reaction chamber; a cathode disposed below the item to be sterilized in the reaction chamber; an injection heater
15 connected to said anode via a mass flow controller ; a plasma power source connected to said cathode via an impedance matching circuit and an impedance matching controller for generating high-frequency power; and a vacuum pump disposed at the lower part of said reaction chamber, wherein an hydrogen peroxide solution in the liquid phase is turned into gaseous hydrogen peroxide by means of said injection heater prior to
20 the sterilization with plasma, and then the gaseous hydrogen peroxide is adjusted and injected at desired pressure by means of said mass flow controller, to accomplish pre-treatment.

According to another aspect of the present invention, an apparatus for supplying a fixed quantity of liquid for generating plasma to said reaction chamber via an
25 discharging pipe comprises: an automatic feeder for feeding automatically an extremely small fixed quantity of liquid for generating plasma via said discharging pipe under the control of the rotation speed of the motor; a vaporizer having a heater connected

to said discharging pipe of said automatic feeder for vaporizing the exhausted liquid; a temperature controller for controlling the temperature of said heater disposed in said vaporizer; and a heater surrounding said exhaust feeding pipe entirely to prevent any condensation of the liquid feeding in said exhaust feeding pipe between the outlet
5 of said vaporizer and said reaction chamber, said heater being connected to said temperature controller which also controls the temperature of said heater.

According to still another aspect of the present invention, an automatic feeder for feeding automatically a fixed quantity of liquid comprises: a direct current motor with a retarder of which the speed is feed-back controlled by a proportional control
10 circuit; a feeding screw connected to a rotating shaft of said direct current motor via a coupling; a supporting member connected mechanically by said feeding screw and a bushing; an injection cylinder for moving linearly by said supporting member; an injection container for sucking and exhausting the liquid as said injection cylinder moves linearly inside said injection container; a feeding valve disposed at the liquid
15 inlet of the injection container, said feeding valve being opened when said injection cylinder extends linearly; a liquid supplying container in which the liquid for generating plasma to be supplied is stored, said liquid supplying container being disposed at said liquid inlet such that the liquid is supplied to said injection container via said feeding valve; a exhaust valve disposed at the outlet of the injection container, said
20 exhaust valve being opened when said injection cylinder retracts linearly, so that the liquid is fed into said discharging pipe; and a displacement sensor disposed at the fixing plate for measuring position of said injection cylinder.

BRIEF DESCRIPTION OF THE DRAWINGS

The preferred embodiment of the present invention will now be described by
25 way of example with reference to the accompanying drawings in which:

Fig. 1 is a schematic view of the low temperature plasma sterilization system

according to the present invention;

Fig. 2 is a schematic view of another embodiment of means for supplying the hydrogen peroxide solution in the liquid phase; and

Fig. 3 is a detailed view of the automatic feeder for feeding automatically liquid shown in Fig. 2.

BEST MODE FOR CARRYING OUT THE INVENTION

Fig. 1 shows a low temperature plasma sterilization system according to an embodiment of the present invention, which is a system for sterilizing an item in the gaseous plasma. The system uses a hydrogen peroxide solution 12 as a source for generation of the plasma to sterilize and disinfect microorganisms on surfaces of an item 9 to be sterilized such as medical instruments, and also uses hydrogen peroxide as active species during generation of the plasma. A pre-treatment is carried out with gaseous plasma prior to the generation of the plasma (which is generated by the electric discharge of gas).

The item 9 to be sterilized such as medical or surgical instruments is wrapped in a packaging material 10. The item 9 wrapped in the packaging material 10 is placed in a reaction chamber 1. In the reaction chamber, an anode 2 is disposed above the item 9 to be sterilized and a cathode 3 is disposed below the item 9 to be sterilized. A mass flow controller 4 is connected to the anode 2 through the reaction chamber 1, while an impedance matching circuit 6 is connected to the cathode 3 through the reaction chamber 1. A vacuum pump 11 is disposed at the lower part of the reaction chamber 1. An injection heater 5 is connected to the mass flow controller 4, and a plasma power source 8 of the RF generator is connected to the impedance matching circuit 6 via the impedance matching controller 7.

The plasma disinfection system of the present invention differs from prior art gas sterilization processes in two important aspects. The first is the use of gaseous

hydrogen peroxide as a precursor of the reactive species rather than an inert gas such as oxygen, nitrogen, etc. The second is that an hydrogen peroxide solution 12 in the liquid phase is turned into gaseous hydrogen peroxide by means of the injection heater 5 prior to the sterilization with plasma, and then the gaseous hydrogen peroxide is adjusted and injected at desired pressure by means of the mass flow controller 4, to accomplish the pre-treatment.

After the item 9 to be sterilized such as medical or surgical instruments, which is wrapped in the packaging material 10 is placed in the reaction chamber 1, the reaction chamber 1 is closed and vacuum is drawn by means of the vacuum pump 11 to remove the gas that is in the chamber. At this time, the hydrogen peroxide solution 12 in the liquid phase is turned into gaseous hydrogen peroxide by means of the injection heater 5, and then the gaseous hydrogen peroxide is adjusted at the pressure of approximately 0.1 to 10 Torr by means of the mass flow controller 4 and injected into the anode 2. The hydrogen peroxide remains in the chamber for a period of sufficient duration to allow the hydrogen peroxide to come in intimate contact with the item 9 to be sterilized, normally approximately 30 minutes.

Then the plasma remains on for up to 50 minutes in the reaction chamber 1 to allow complete sterilization, although sterilization can be effective in periods as short as 5 minutes from initial plasma generation, depending on the plasma power source 8 that is applied to the cathode 3 and the concentration of the hydrogen peroxide.

At this time, the plasma is generated as follows: The power is set to the desired level using the plasma power source 8, and then pass through the impedance matching circuit 6 using the impedance matching controller 7 so that the level corresponds to resistance value of the gaseous hydrogen peroxide in the reaction chamber 1, resulting in that the optimal power is supplied to the cathode 3.

Therefore, it is preferable to apply the optimal power in order to obtain the optimal efficiency of sterilization since the efficiency of sterilization of the present

invention relies on the plasma power source 8 as well as the concentration of hydrogen peroxide. The item 9 to be sterilized is wrapped in the packaging material 10 and then place in the reaction chamber 10. The preferred packaging materials are fibriform polyethylene packaging material commonly available under the trademark "TYVEK" of fibriform polyethylene terephthalate packaging material commonly available under the trademark "MYLAR". Paper packaging materials may also be used. With the paper packaging, however, longer processing times may be required to achieve sterilization because of possible interactions of hydrogen peroxide and other reactive species with the paper.

10 The low temperature plasma sterilization system generates the plasma with the temperature of less than 100°C. The plasma is generated at the pressure of the gaseous hydrogen peroxide as the reaction gas of less than 10 Torr. The plasma power source 8 is of a high-frequency (RF 13.56 MHz) capacity combination type in which the high-frequency power is applied intermittently in the form of pulse.

15 In the present invention, the intermittent application is employed in order to prevent the overheating of the reaction gas in the reaction chamber 1 as well as to prevent the overheating of the item 9 to be sterilized. With the intermittent application, high-frequency power would be applied for 0.5 milliseconds and then turned off and applied again 1.0 milliseconds later.

20 As mentioned above, hydrogen peroxide is injected into the anode 2 of the reaction chamber 1 in order to carry out the pre-treatment. At this time, the concentration of the gaseous hydrogen peroxide is 0.05 to 10 mg/liter. The higher concentration of hydrogen peroxide will result in shorter sterilization times since the efficiency of the sterilization becomes higher.

25 The minimum concentration of hydrogen peroxide injected into the reaction chamber 1 is approximately 0.125 mg/liter. Auxiliary gases such as oxygen, nitrogen, argon or the like may be added into the reaction chamber with the hydrogen peroxide

when the hydrogen peroxide is injected at the appropriate concentration.

The general operation of the low temperature plasma sterilization system of the present invention constructed as described above is as follows:

(1) The door of the reaction chamber 1 is opened so that the reaction chamber
5 is under the atmospheric pressure.

(2) The item 9 to be sterilized such as medical or surgical instruments is put into a wire netting container 13, and then wrapped in the packaging material 10.

(3) the wire netting container 13 wrapped in the packaging material 10 is put into the reaction chamber 1 and then the door of the chamber is closed.

10 (4) The reaction chamber 1 is evacuated to a pressure of up to 10^{-3} Torr using the vacuum pump 11.

(5) A hydrogen peroxide solution in the liquid phase is injected at a fixed quantity into the anode 2 using the mass flow controller 4 from the injection heater 5 such that the pressure in the reaction chamber 1 is maintained between 0.5 and
15 10 Torr. At this time, the concentration of the hydrogen peroxide injected into the reaction chamber 1 may be from 0.05 to 10 mg/liter, preferably 0.208 mg/liter.

The above process is a pre-treatment which is carried out for the time of from 5 to 30 minutes. The duration of the pre-treatment time may depend on the type of packaging material used, the number of items 9 to be sterilized, and the placement
20 of the items in the reaction chamber.

(6) After the pre-treatment is completed, the high-frequency power from the plasma power source 8 is applied to the cathode 3 in the reaction chamber 1 via the impedance matching controller 7 and the impedance matching circuit 6, to generate the plasma.

25 (7) The plasma power source 8, which is a high-frequency power required to generate the plasma, may be employed as a continuous application type. Alternatively, pulsed high-frequency power with the frequency of 1 KHz may be used in order

to prevent the overheating of the reaction gas as well as the overheating of the item 9 to be sterilized. The item 9 remains in the plasma for a period of from 5 to 60 minutes to effect complete sterilization.

(8) After the sterilization is completed in the reaction chamber 1, the plasma
5 power source 8 is removed, the pressure in the reaction chamber 1 is subject again to the atmosphere pressure, and the sterilized item 9 wrapped in the packaging material 10 is taken out, to complete all the process.

When the item 9 to be sterilized such as medical or surgical instruments is
sterilized with the present invention, no additional steps are required to remove residual
10 hydrogen peroxide from the sterilized item 9 or its packaging material 10, since the hydrogen peroxide is decomposed into non-toxic products during the plasma treatment unlike the ethylene oxide process, which is a prior gas sterilization process.

Fig. 2 is a schematic view of another embodiment for supplying a extremely
small fixed quantity of the hydrogen peroxide solution in the liquid phase into the
15 reaction chamber.

An automatic feeder 20 for feeding automatically an extremely small fixed
quantity of liquid for generating plasma is provided at an discharging pipe 40 thereof
with a vaporizer 24 having a heater mounted therein. To the heater (26) is connected
to a temperature controller 25 which controls the temperature of the heater 26 by
20 the proportional control.

The automatic feeder 20 for feeding automatically a fixed quantity of liquid
enables an injection cylinder 45 connected to a reduction motor 31 of which the speed
is feed-back controlled by a proportional control circuit 12 of the controller for rotation
speed of the motor to move linearly at the desired speed, as shown in Fig. 3. As
25 a result, it is possible to control amount of supply per unit time, which is determined
by a ratio of the cross section of the injection cylinder 45 to the cross section of
the outlet from which the solution is discharged.

In other words, after the liquid whose volume corresponds to that of an injection container 46 is used out, the motor 31 is rotated reversely allowing the liquid from a liquid supplying container 48, which is disposed above the injection container 46 and connected to the injection container 46, to be injected automatically into the injection container 46 along a liquid feeding pipe 38, which is connected to the other side of the injection container 46. The supplied liquid is guided to the vaporizer 24 whose temperature is controlled automatically by a proportional temperature control circuit 42 of the temperature controller 25, and heated by the heater 26 in the vaporizer 24. The heated liquid is turned into gas, and then supplied into the reaction chamber 1. In the reaction chamber 1, plasma is generated from the supplied gas which is used to carry out the sterilization and disinfection process.

The automatic feeder 20 for feeding automatically an extremely small fixed quantity of liquid will now be described in more detail.

The direct current motor 31 with a retarder 32 of which the speed is feed-back controlled by the proportional control circuit 42 has a rotating shaft connected to a coupling 33. To the other side of the coupling 33 is connected a feeding screw 36.

The feeding screw 36 is connected mechanically to a supporting member 35 via a bushing 34. To the supporting member 35 is connected the injection cylinder 45 which moves linearly. If the injection cylinder 45 moves linearly, the liquid contained in the injection container 46 is discharged into an discharging pipe 40 through an exhaust valve 44. At this time, the injection container 46 is fixed by means of fixing plates 37 and 39, which prevents the injection container 46 from moving during the linear movement.

After the liquid contained in the injection container 46 is used out, the motor 31 is rotated reversely allowing the liquid from the liquid supplying container 48, which is disposed above the injection container 46 and connected to the injection container 46, to be injected into the injection container 46 via through the liquid

feeding pipe 38 and the feeding valve 43.

The transfer distance per unit time of the injection cylinder 45 may be derived from the ratio of the cross section of the exhaust valve 44 to the cross section of the inside of injection container 46, and the rotation speed per unit time of the direct
5 current motor 31 with the retarder 32 may be derived from the transfer distance. As a result, the time required for supplying the whole liquid into the injection container 46 may be yielded. Consequently, it is possible to supply automatically the injected liquid at an extremely small fixed quantity.

The feed-back control of the proportional control circuit 42 is used in order
10 to control the speed of the direct current motor 31 with the retarder 32. The position of the injection cylinder 45 is measured by a displacement sensor 49 disposed at the fixing plate 37 for fixing the injection container 46.

The assembled supplying components are mounted as a whole on a mounting member 41. The liquid from the automatic feeder 20 for feeding automatically an
15 extremely small fixed quantity of liquid is supplied into the vaporizer 24 via the exhaust feeding pipe 23. The supplied liquid is vaporized in the gas phase by the heater 26 mounted in the vaporizer 24, and then injected into the reaction chamber 1. At this time, the temperature of the heater 26 inside the vaporizer 24 is controlled by the temperature control circuit 25 of the proportional control type.

20 When the liquid is supplied, it may be condensed in the exhaust feeding pipe 23 due to difference of the temperature between the exhaust feeding pipe 23 and the supplied liquid. In order to prevent the condensation, a heater 22, which is in the form of sheet and also connected to the temperature controller 25, is disposed around and therefore surrounds the exhaust feeding pipe 23 entirely to prevent any
25 condensation of the vaporized liquid. Plasma is generated from the supplied gas to be used in the plasma sterilization process or the semiconductor deposition process.

Example 1

The following example shows the results of the sterilization process in the plasma sterilization system. Please note that O indicates the number of organisms before the sterilization and T indicates the number of organisms after the sterilization.

5 Table 1 shows the results of the sterilization process in the ozone/hydrogen peroxide plasma sterilization system. The test was carried out under the conditions that the concentration of the hydrogen peroxide is 0.2 mg/liter, the pressure is 1.5 Torr, the high-frequency pulse power is 150 W, and the total processing time is 15 minutes.

10 Please note that all the tests fulfilled the pre-treatment for 10 minutes in the reaction gas (1.5 Torr) shown in Table 1.

[Table 1]

RESULTS OF THE STERILIZATION PROCESS IN THE OZONE/HYDROGEN
PEROXIDE PLASMA STERILIZATION SYSTEM

Reaction Gas	Results of the Process (T/O)
O ₂	$9.1 \times 10^5 / 1.3 \times 10^5 = 0.72$
H ₂ O ₂	$0/3.4 \times 10^5 = 0$

15 From Table 1, it can be seen that the efficiency of the sterilization process is higher if the hydrogen peroxide plasma is used rather than if the ozone plasma is used.

Example 2

20 The test was carried out in order to verify the efficiency of the sterilization process in the same hydrogen peroxide plasma sterilization system depending on the concentration of the hydrogen peroxide (mg H₂O₂/liter). The results of the test are given in Table 2.

[Table 2]

EFFICIENCY OF STERILIZATION PROCESS DEPENDING ON THE
CONCENTRATION OF HYDROGEN PEROXIDE

Concentration (mg H ₂ O ₂ /liter)	H ₂ O ₂ Alone (T/O)	H ₂ O ₂ + Water Vapor (T/O)
0.1	1.0	1.0
0.2	1.0	1.4×10^{-2}
0.6	9.1×10^{-2}	0

- 5 The results of Table 2 illustrate that the efficiency of the sterilization process is the highest if the concentration of the hydrogen peroxide is 0.6 and that the efficiency of the sterilization process is higher if the hydrogen peroxide and water vapor plasma is used rather than if the hydrogen peroxide plasma alone is used.

Example 3

- 10 The test was carried out in order to verify the efficiency of the sterilization process in the same plasma sterilization system depending on the reaction pressures (Torr) of air, hydrogen peroxide, and a mixture of air and hydrogen peroxide. The results of the test are given in Table 3.

[Table 3]

- 15 EFFICIENCY OF STERILIZATION PROCESS DEPENDING ON THE
REACTION PRESSURE OF HYDROGEN PEROXIDE

Pressure (Torr)	Air Plasma Alone (T/O)	H ₂ O ₂ Plasma Alone	H ₂ O ₂ + Air Plasma
0.5	6.0×10^{-1}	9.6×10^{-1}	4.1×10^{-1}
1.0	6.7×10^{-1}	1.0	1.4×10^{-2}
1.5	2.8×10^{-1}	3.9×10^{-1}	0
2.0	2.4×10^{-1}	6.6×10^{-1}	1.9×10^{-4}

The results of Table 3 illustrate that the efficiency of the sterilization process is higher if the hydrogen peroxide and air plasma is used rather than if the air plasma alone is used or the hydrogen peroxide plasma alone is used and that the efficiency of the sterilization process is the highest if the pressure of the reaction gas is 1.5 Torr.

Example 4

The test was carried out in order to verify the efficiency of the sterilization process in the same plasma sterilization system depending on the plasma generation power (Watt) to a mixture plasma of air and hydrogen peroxide and air plasma alone.

The results of the test are given in Table 4.

[Table 4]

EFFICIENCY OF STERILIZATION PROCESS DEPENDING ON
HIGH-FREQUENCY POWER

Power (Watt)	Air Plasma Alone (T/O)	H ₂ O ₂ + Air Plasma (T/O)
0	1.0	4.0×10^{-1}
50	4.0×10^{-1}	8.1×10^{-2}
100	6.7×10^{-1}	2.5×10^{-3}
150	2.4×10^{-1}	0

The results of Table 4 illustrate that the efficiency of the sterilization process is higher if the hydrogen peroxide and air plasma is used rather than if the air plasma alone is used and that the efficiency of the sterilization process is the highest if the high-frequency power, which is the power required for generating the plasma, is 150 Watts.

INDUSTRIAL APPLICABILITY

With the plasma disinfection system according to the present invention as

described above, hydrogen peroxide is employed as a precursor of the active species and provided is an initial contact of surfaces of the material to be sterilized with the gaseous hydrogen peroxide injected into a reaction chamber as a pre-treatment step before the generation of plasma at a power level sufficient to achieve sterilization, 5 resulting in that the total time and power required to accomplish sterilization with the low temperature plasma, the use of the pre-treatment with hydrogen peroxide also allows sterilization to occur within many different types of packaging material, and no toxic residues remain on the sterilized items after plasma treatment since the decomposition products of hydrogen peroxide in plasma, which is obtained when final 10 sterilization is carried out with the hydrogen peroxide plasma generated by the application of the power after the pre-treatment, include water, oxygen and hydrogen.

Furthermore, an extremely small quantity of the liquid can be supplied into the sterilization chamber or the reaction chamber by means of the automatic liquid feeder with the simple construction in the plasma sterilization system used for the 15 purpose of sterilization and disinfection in hospitals or the liquid supplier for supplying liquid for generation of vapor of the metal used frequently in the semiconductor process, resulting in that the costs for manufacturing the apparatus for supplying an extremely small quantity of the liquid in the in the plasma sterilization system or the liquid supplier for supplying liquid for generation of vapor of the metal.

What is claimed is:

1. A plasma disinfection system comprising: a reaction chamber (1) for receiving an item (9) to be sterilized, said item being wrapped in a packaging material (10); an anode (2) disposed above the item (9) to be sterilized in the reaction chamber (1); a cathode (3) disposed below the item (9) to be sterilized in the reaction chamber (1); an injection heater (5) connected to said anode (2) via a mass flow controller (4); a plasma power source (8) connected to said cathode (3) via an impedance matching circuit (6) and an impedance matching controller (7) for generating high-frequency power; and a vacuum pump (11) disposed at the lower part of said reaction chamber (1), wherein an hydrogen peroxide solution (12) in the liquid phase is turned into gaseous hydrogen peroxide by means of said injection heater (5) prior to the sterilization with plasma, and then the gaseous hydrogen peroxide is adjusted and injected at desired pressure by means of said mass flow controller (4), to accomplish pre-treatment.
2. The system of claim 1, wherein said plasma is generated at the pressure of the gaseous hydrogen peroxide as the reaction gas of less than 10 Torr, and said plasma power source (8) is of a high-frequency (RF 13.56 MHz) capacity combination type in which the high-frequency power is applied intermittently in the form of pulse.
3. The system of claim 1, wherein the concentration of the gaseous hydrogen peroxide ranges between 0.05 and 10 mg/liter.
4. The system of claim 1, wherein means for supplying liquid for generating plasma to said reaction chamber (1) comprises: an automatic feeder (20) for feeding automatically an extremely small fixed quantity of liquid for generating plasma via said discharging pipe (40) under the control of the rotation speed of the motor (31);

a vaporizer (24) having a heater (26) connected to said discharging pipe (40) of said automatic feeder (20) for vaporizing the exhausted liquid; a temperature controller (25) for controlling the temperature of said heater (26) disposed in said vaporizer (24); and a heater (26) surrounding said discharging pipe entirely to prevent any condensation of the liquid feeding in said discharging pipe between the outlet of said vaporizer (24) and said reaction chamber (1), said heater (26) being connected to said temperature controller (25) which also controls the temperature of said heater (26).

5. The system of claim 4, wherein said automatic feeder (20) for feeding automatically a fixed quantity of liquid comprises: a direct current motor (31) with a retarder (32) of which the speed is feed-back controlled by a proportional control circuit; a feeding screw (36) connected to a rotating shaft of said direct current motor (31) via a coupling (33); a supporting member (35) connected mechanically by said feeding screw (36) and a bushing (34); an injection cylinder (45) for moving linearly by said supporting member (35); an injection container (46) for sucking and exhausting the liquid as said injection cylinder (45) moves linearly inside said injection container; a feeding valve (43) disposed at the liquid inlet of said injection container (46), said feeding valve (43) being opened when said injection cylinder (45) extends linearly; a liquid supplying container (48) in which the liquid for generating plasma to be supplied is stored, said liquid supplying container (48) being disposed at said liquid inlet such that the liquid is supplied to said injection container (46) via said feeding valve (43); an exhaust valve (44) disposed at the outlet of the injection container (46), said exhaust valve (44) being opened when said injection cylinder (45) retracts linearly, so that the liquid is fed into said discharging pipe (40); and a displacement sensor (49) disposed at the fixing plate (37) for measuring position of said injection cylinder (45).

6. A plasma disinfection system comprising the steps of injecting a hydrogen peroxide solution (12) in the liquid phase at a fixed quantity into a reaction chamber (1) using a mass flow controller (4) from an injection heater (5) such that the pressure in the reaction chamber (1) is maintained between 0.5 and 10 Torr as pre-treatment;

5 applying high-frequency power from a plasma power source (8) to a cathode (3) in the reaction chamber (1) via an impedance matching controller (7) and an impedance matching circuit (6) to generate the plasma; and

 maintaining a sterilized item (9) in the plasma for a period of from 5 to 60 minutes to effect complete sterilization.

10 7. The system of claim 6, wherein the plasma power source (8), which is a high-frequency power required to generate the plasma, is employed as a continuous application type or pulsed high-frequency power with the frequency of 1 KHz is used in order to prevent the overheating of the reaction gas as well as the overheating of the item (9) to be sterilized.

FIG. 1

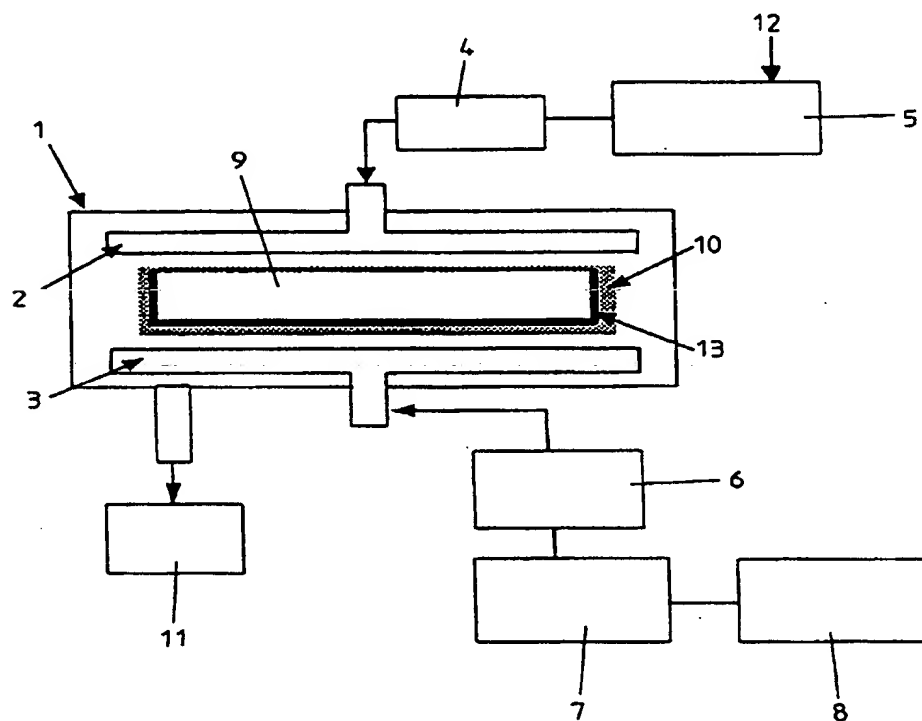
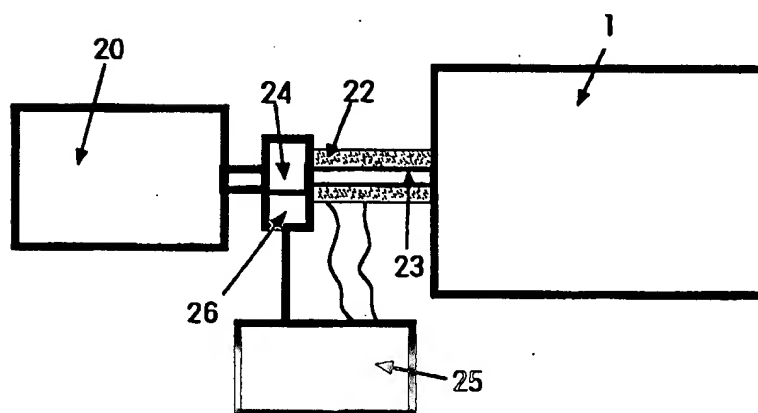
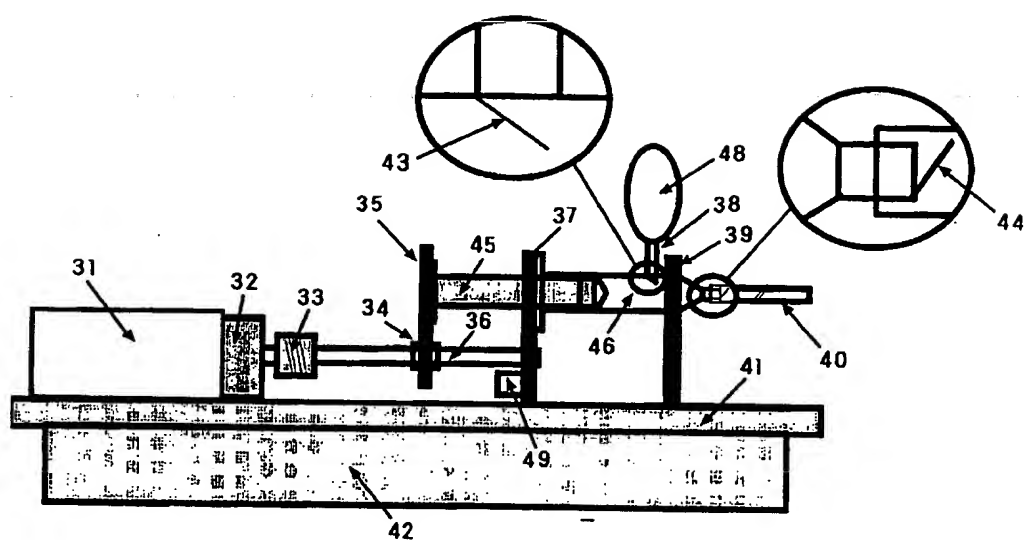


FIG. 2



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FIG. 3



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR00/00539

A. CLASSIFICATION OF SUBJECT MATTER IPC7 A61L 2/14, A61L 2/18, A61L 2/24 According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC7 A61L 2/14, A61L 2/18, A61L 2/20, A61L 2/24 Documentation searched other than minimum documentation to the extent that such documents are included in the files searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) NPS, IPN, WIPS		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4756882 A (SURGIKOS INC.) 12 JANUARY 1988 see column 2; line 67-column 3; line 14 column 4; line 59-column 5; line 3 column 5; line 50-column 6; line 28 see example V	1-3, 6-7
<input type="checkbox"/> Further documents are listed in the continuation of Box C.		
<input checked="" type="checkbox"/> See patent family annex.		
<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&" document member of the same patent family</p>		
Date of the actual completion of the international search 27 DECEMBER 2000 (27.12.2000)		Date of mailing of the international search report 28 DECEMBER 2000 (28.12.2000)
Name and mailing address of the ISA/KR Korean Industrial Property Office Government Complex-Taejon, Dunsan-dong, So-ku, Taejon Metropolitan City 302-701, Republic of Korea Facsimile No. 82-42-472-7140		Authorized officer KIM, Seung Soo Telephone No. 82-42-481-5581

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/KR00/00539

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4756882 A	12.01.88	AT 104864 E	15.05.94
		AU 616648 B2	07.11.91
		BR 8800301 A	06.09.88
		CA 1299835 A1	05.05.92
		DE 3889235 T2	06.10.94
		EP 278623 B1	27.04.94
		ES 2063029 T3	01.01.95
		IE 62878 B	08.03.95
		KR 9710057 B1	20.06.97
		NZ 223182 A	26.04.90
		ZA 8800531 A	27.09.89